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August 18, 2008

Mr. Sam Chummar
Remedial Project Manager
U.S. EPA Region 5 (SR-6J)
77 West Jackson Boulevard
Chicago, IL 60604-3590



Subject: Plainwell Mill Site, Plainwell, Michigan
(Operable Unit #7 of the Allied Paper, Inc./Portage Creek/Kalamazoo River
Superfund Site)
Standard Operating Procedures (SOP), Multi-Area QAPP

Dear Sam,

As we discussed, Weyerhaeuser is submitting the applicable and already reviewed Standard Operating Procedures (SOP) for use with the Multi-Area Quality Assurance Project Plan (QAPP). This QAPP is being revised for the Test Pit Investigation associated with draft investigation activities planned for the Plainwell Mill (Operable Unit 07) on the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site. The selected SOPs provided for your consideration are primarily associated with the soil analyses required for the test pit investigation. A second set of modified SOPs that addresses additional media will be sent tomorrow. These documents have been sent for your review and further direction regarding how to expedite the QAPP review process and allow investigation activities to begin during September or October.

The table attached to this letter summarizes the responses to comments that have been incorporated into each SOP as well as an explanation as to how a comment or suggestion will be addressed or why the SOP has not been modified. Most of the comments are addressed in this tabular summary. However, there are a few comments that are more general and those have been addressed in this letter.

Applicable General SOP comments:

1. SOPs from the WATS laboratory have been expired. During the QAPP review time please submit the latest copy of all SOPs which plan to be used for this project.

The latest copy of all the WATS SOPs for this project are attached to this cover letter and are no longer labeled as expired.

10.1.a. CAS should provide SOP on how they identify and segregate their 'waste' streams.

The section listed is not intended to identify the RCRA waste and PCB waste characteristics. It only mentions those types of wastes for readability and clarification. No changes are planned.

10.1.b. The CAS documents submitted have a box for a Document Control Number on the title sheets. The boxes were not completed and no indication was present that the document was an uncontrolled copy.

The lack of color on the page is the indication that these methods are uncontrolled copies

10.1.c. Weyerhaeuser's 2 AM E SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP AM E-8082 includes a section titled 'REPORT' which identifies how many significant figures should be used in reporting the data and a section titled 'Key Words.' These sections are not included in SOP AM E-8270. Precision and accuracy discussion is included in Section 10: Quality Control in SOP AM E-8270 but included in Section 11: Report in SOP AM E-8082.

WATS Quality Management System (QMS) has a template for writing methods. Our method-writing SOP notes that this template is a guideline. Additionally, the EPA's SOP for writing SOPs (EPA QA/G-6) is also a guideline, not a requirement. Therefore, our methods are not required to have the same format. However, the Quality Team of WATS discussed this issue and concluded that it is a good idea for all of their methods to have the same format (same headings and types of information under those headings). WATS has recently revamped their QMS and plans to adjust SOP formats during their regular SOP review schedule.

10.1.d. Weyerhaeuser's 3 AM G SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP AC G-3550 includes sections titled 'Estimate of Preparatory Time' and 'Amount of Sample' not included in the other 2 SOPs. SOP AC G-3550 does not include a section titled 'Key Words' section. SOP AC G-3520 does not include sections titled 'Sample Handling and Storage' or 'References.'

See response to comment 10.1.c.

10.1.e. Columbia's 3 SMO SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP SMO-GEN does not include a section titled Definitions. SOP SMO-SDIS does not include a section titled Discussions.

The three SMO SOPs contain the necessary sections to provide work instructions. Certain sections are not necessary for some non-analytical procedures (e.g. SMO-GEM having a discussions section). These three SOPs meet the standards set by NELAP and have been reviewed and approved by their NELAP accreditation body and other certifying agencies.

Mr. Sam Chummar

August 18, 2008

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Specific SOP Comments:

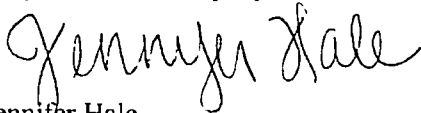
The table attached provides a summary of the current status of the SOPs including updates to specific SOPs in response to comments made by EPA to Weyerhaeuser on June 27, 2007. The table does not include response to comments made regarding the MDEQ SOP #213 Soil Fraction Preparation for Lead Analysis, Revision 2, Effective Date 01/05. This SOP is controlled by MDEQ, and therefore can not be modified by Weyerhaeuser or RMT.

Please call me or Kathy Huibregtse if you have any questions.

Thank you for your prompt attention to this matter.

Sincerely,

Weyerhaeuser Company

A handwritten signature in cursive script that reads "Jennifer Hale".

Jennifer Hale
Environmental Manager

Enclosures

Cc: Martin Lebo-Weyerhaeuser
Kathy Huibregtse- RMT
James Hutchens- RMT

QAPP SOP Revision Summary

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	SUMMARY OF RESPONSE TO COMMENTS
W-1	Organochlorine and PCB (aroclor) pesticide analysis by GC/ECD, AM E-608, June 20, 2003	Already approved by EPA with last version of the Multi-Area QAPP.
W-2	Suspended solids in water and wastewater. AM E-160.2, April 24, 2002	Already approved by EPA with last version of the Multi-Area QAPP.
W-3	Total and total soluble phosphorus in water, AM E-365.3, August 11, 2004	Already approved by EPA with last version of the Multi-Area QAPP.
W-4	Polychlorinated biphenyl analysis by GC/ECD, AM E-8082, June 20, 2003	Already approved by EPA with last version of the Multi-Area QAPP.
W-5	Daily balance, water, and temperature checks, AQ-O-DLYCHK, July 7, 2005	Already approved by EPA with last version of the Multi-Area QAPP.
W-6	General Procedures for Performing Chromatographic Separations and Calibrations, AL G-8000, July 3, 2003	Already approved by EPA with last version of the Multi-Area QAPP.
W-7	Sample Management Procedures, OP S-SMO, September 5, 2006	Already approved by EPA with last version of the Multi-Area QAPP.

QAPP SOP Revision Summary

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	SUMMARY OF RESPONSE TO COMMENTS
W-8	Procedure for Training Personnel to Perform Test Methods and Standard Operating Procedures. OQ TRAIN. June 1, 2003.	Already approved by EPA with last version of the Multi-Area QAPP.
W-9	Analytical Procedure 3500, AC G-3500, July 6, 2003	Per EPA June 2007 comment 10.6, WATS updated the referenced SW 846 method series numbers.
W-10	Extraction by Continuous Liquid-Liquid Extractor, AC G-3520, April 19, 2004.	Per EPA June 2007 comment 10.8, WATS added a note regarding the use of Millipore water to the reagents section. The storage and handling of extracted analytes is discussed in the corresponding method SOPs.
W-11	Sonication for Soil and Sediments, AC G-3550, December 7, 1993	Per EPA June 2007 comment 10.7, WATS updated SOP references (new SOPs are included as W-16, 18 and 19), and added container details to Section 9.3.4. Sections 9.3.1 and 9.4.3 state that chromatography tracking sheets are used to determine volumes of surrogates and matrix spike solutions and this information is logged in the lab standard log book.
W-12	Acid Digestion of Aqueous Samples & Extracts for Metals Analysis by ICP & ICPMS, AM E-3010, March 17, 2003	Per EPA June 2007 Comment 12, SOP meets technical requirements of SW 846 Method 3010. WATS will consider the suggestion with this comment during the next SOP revision.
W-13	Acid Digestion of Sediments, Soils, Sludges, and Other Solid Samples for Analysis by ICP & ICPMS, AM E-3050, March 17, 2003	Per EPA June 2007 Comment 13, SOP meets technical requirements of SW 846 Method 3050. WATS will consider the Suggestion with this comment during the next SOP revision.
No longer in use	Standard Operating Procedure for Perkin-Elmer ELAN 5000 ICPMS, AI M-ICPMS, March 25, 2003	Discontinued when the ELAN 5000 ICPMS was taken out of service; replaced by AM E-200.8M (W-14). A separate SOP for method 6020 was not created because WATS does not use this 6020 for metals analysis.

QAPP SOP Revision Summary

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	SUMMARY OF RESPONSE TO COMMENTS
No longer in use	Metals Team Supplement QA/QC Guidelines to the A&T. AQ M-METALS.	Per EPA June 2007 Comment 16, AQ M-METALS has been phased out. QC requirements are now specified in the SOPs for the specific method.
W-14	Metals Analysis by ICPMS. AM E-200.8M. May 31, 2006	WATS rewrote this SOP per EPA June 2007 comment 15 to include the preparation procedures for EPA Method 200.8.
W-15	Capillary GC/MS for Semivolatile Organics. AM E-8270. July 20, 2007	Per EPA June 2007 comment 10.10, the SOP referenced in Section 8.2.1 was added to the QAPP SOP list (W-20).
W-16	Total Solids in Solid and Semi-Solid Samples. AM S-2540G. December 3, 2002.	SOP AM S-2540G was added per U.S. EPA June 2007 comment 10.7.b.
W-17	Procedure for Florisil Clean Up of Organic Extracts. AC G-3620. June 20, 2003.	SOP AC G-3620 was added due to reference in SOP AM E-8082.
W-18	Gel Permeation Clean-Up. AC G-3640. May 15, 2002.	SOP AC G-3640 was added per EPA June 2007 comment 10.7.a.
W-19	Condensation and Evaporation Techniques. AC G-CONC. June 23, 2003.	SOP AC G-CONC was added per U.S. EPA June 2007 comment 10.7.c.

QAPP SOP Revision Summary

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	SUMMARY OF RESPONSE TO COMMENTS
W-20	Organic Laboratory Analytical Standards. AC G-ORGSTD. July 17, 2007.	AC G-ORGSTD was added per EPA June 2007 Comment 10.10.
C-1	Sample Disposal. SMO-SDIS, Rev 7 March 26, 2008	This is an updated version, which includes the attachment per EPA June 2007 Comment 10.2a, and edits per Comments 10.2c, d, and f. No changes have been made regarding comments 10.2 b,e,g or h since this SOP is not intended to identify the characteristics of RCRA or PCB wastes (b) , and the sample disposal procedures have been reviewed and approved by the Washington Department of Ecology (Comments 10.2 e,g, and g).
C-2	Sample Tracking and Internal Chain-of-Custody. SMO-SCOC, Rev 11, March 21, 2008.	This is an updated version, which incorporates the suggested change per EPA June 2007 Comment 10.3a.
C-3	Sample Receiving. SMO-GEN, Rev 24, March 5, 2008	This is an updated version of the SOP. As per EPA June 2007 Comment 10.4, the SOP has been revised to include the sample preservation table by reference (10.4 a). Changes have been incorporated to establish that any liquid in a shipping cooler from a broken sample will be handled as a potentially hazardous waste (10.4 b). Finally, the accepted industry-wide practices for signing COCs and associated COC procedures have been reviewed and approved by the applicable NELAP accreditation body (10.4c).
C-4	Mercury in Water by Atomic Fluorescence Spectrometry. MET-1631	Per EPA June 2007 Comment 21, SOP meets technical requirements of EPA method 1631.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, ILLINOIS 60604

REPLY TO THE ATTENTION OF: SR-6J

VIA ELECTRONIC MAIL AND USPS

Wednesday, June 27, 2007

Linda E Hicken
RMT, Inc
744 Heartland Trail
Madison, Wisconsin 53717-1934

RE: Portage Creek/Kalamazoo River Site
Plainwell Mill
Preliminary Comments: Quality Assurance Project Plan

Dear Ms. Hicken:

In September 2006, RMT, Inc. provided a *Draft Quality Assurance Project Plan (QAPP)* on behalf of Weyerhaeuser Company, in accordance with the *Consent Decree For The Design And Implementation Of Certain Response Actions At Operable Unit #4 And The Plainwell Inc. Mill Property Of The Allied Paper, Inc/Portage Creek/Kalamazoo River Superfund Site (CD)*. Weyerhaeuser and RMT have indicated a change in the format, approach, and scope of the QAPP at the Plainwell Mill. To aid in the expedient revision and quality of revision, the Region 5 is providing preliminary comments and recommendations regarding the draft QAPP, dated September 2006.

1. SOPs from the WATS laboratory have been expired. During the QAPP review time please submit the latest copy of all SOPs which plan to be use for this project.
2. Page 44 of 61. Paragraph 2.10 Data Management Chapter 2.10.2 Laboratory Data Reduction Procedure. Please clarify the definition for the Level 1, 2 and 3 data review.
3. Table 2-2. Summary of Analytical Program for the Groundwater Investigation. The number of samples which will be collected for the all planed analytes (VOCs, PAHs, PCBs, Metals and TSS) are one (1). Do you plan to collect MS/MSD, field blank, field duplicate field equipment blank, trip blank, etc.?
4. Page 6 of 61. U.S. EPA Region 5 QA/QC Coordinator should be changed to U.S. EPA Region 5 QAPP reviewer.

5. Please provide a schedule of work to be performed in graphic or tabular format. The time line must include the start and completion dates for all project activities. (Page 15 of 68 "Instruction on the preparation of Superfund Division Quality Assurance Project Plan," revision 0, June 2000).
6. Which exact Operable Unit will be tested? In the QAPP Units #4, 6 and 7 were found.
7. Submit Chain-of-Custody forms from all laboratories which will participate in the project.
8. Provide the Project/Task Organization Chart during the QAPP review time.
9. The Field/Sampling plan should be more specific. SOPs for field parameters should be submitted.
10. A total of sixteen standard operating procedures for inclusion in the Allied Paper QAPP were reviewed for technical accuracy and agreement with published references. Eleven SOPs are authored by Weyerhaeuser Analytical & Testing Laboratory (WATS); four SOPs are authored by Columbia Analytical Services (CAS) and one SOP is authored by Michigan Department of Environmental Quality (MDEQ). The following comments discuss the findings of these reviews.

10.1. General Comments:

- a. CAS should provide SOP on how they identify and segregate their 'waste' streams.
- b. The CAS documents submitted have a box for a Document Control Number on the title sheets. The boxes were not completed and no indication was present that the document was an uncontrolled copy.
- c. Weyerhaeuser's 2 AM E SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP AM E-8082 includes a section titled 'REPORT' which identifies how many significant figures should be used in reporting the data and a section titled 'Key Words'. These sections are not included in SOP AM E-8270. Precision and accuracy discussion is included in Section 10: Quality Control in SOP AM E-8270 but included in Section 11: Report in SOP AM E-8082.
- d. Weyerhaeuser's 3 AM G SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP AC G-3550 includes sections titled 'Estimate of Preparatory Time' and 'Amount of Sample' not included in the other 2 SOPs. SOP AC G-3550

does not include a section titled 'Key Words' section. SOP AC G-3520 does not include sections titled 'Sample Handling and Storage' or 'References'.

- e. Columbia's 3 SMO SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP SMO-GEN does not include a section titled Definitions. SOP SMO-SDIS does not include a section titled Discussions.

10.2. Standard Operating Procedure for Sample Disposal (SOP No.: SMO-SDIS, Rev 6, Feb 21, 2006) submitted by Columbia Analytical Services, Inc. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 5.1.2.2 references Attachment I – Potentially Hazardous Materials Criteria, but no copy of the attachment was submitted with the SOP.
- b. Section 5.1.2.3 does not identify the distinguishing characteristics between 'RCRA' waste and 'PCB' waste.
- c. Section 5.2.2 – aqueous samples are held for 31 days but 'solid' samples are held for 2 months. Each month may be either 28 (29 on a leap year), 30 or 31 days long. It would be preferable if the SOP lists a specific number, such as 60 days.
- d. Section 5.2.3.1 indicates that disposal of non-hazardous preserved samples are flushed down the drain with large amounts of water. Preserved samples should be neutralized prior to disposal as preserved samples (acidified metals samples and base preserved cyanide samples are hazardous by pH).
- e. Section 5.2.3.2/5.2.3.3 indicate that solid samples considered non-hazardous are disposed of in the garbage. It is not clear how much testing is done on the samples to determine if any hazardous constituents are present. Since there may be hazardous constituents present which were not tested for, solids should be drummed or lab packed and disposed of as potentially hazardous.
- f. Section 5.2.3.4 does not contain any reference to checks of hazardous constituents other the odor. Foul Condensates should be checked for other hazardous constituents. Also, no check is made to insure that organosulfides are sufficiently neutralized/broken down. The bleach solution pH should then be checked to insure that it is within the correct range for discharge.

- g. Section 5.2.6.2 indicates that containers for non-hazardous samples are dumped in the normal laboratory garbage. Those which contained preserved samples should be triple rinsed and the rinsate neutralized to insure no preservative residue is present in the bottles.
 - h. Section 5.2.6.3 indicates that glass containers which contained hazardous samples are included in the sample waste handling but that plastic containers are dumped in the normal laboratory garbage. Plastic containers should at least be triple rinsed and the rinsate treated as hazardous liquid waste or the plastic bottles treated as hazardous solid waste.
- 10.3. Standard Operating Procedure for Sample Tracking and Internal Chain-of-Custody (SOP No.: SMO-SCOC, Rev 10, March 21, 2006) submitted by Columbia Analytical Services, Inc. The SOP was reviewed for technical accuracy and agreement with published references.
- a. Section 6.5 indicates that, when disposal is performed, each container is scanned. No mention is made regarding how the custody of hazardous samples is handled.

Suggestion:

Include scanning into hazardous waste drum or lab pack.

- 10.4. Standard Operating Procedure for Sample Receiving (SOP No.: SMO-GEN, Rev 22, October 7, 2005) submitted by Columbia Analytical Services, Inc. The SOP was reviewed for technical accuracy and agreement with published references.
- a. Sec 5.1.1 A copy of Table 8-1 from the CAS Quality Assurance Manual should be part of SOP. Does this table indicate a nominal amount of acid or base to be added to the sample in order to preserve the sample properly?
 - b. Section 5.1.8.3 Liquid in coolers from broken samples should be treated as potentially hazardous and disposed of accordingly. Since sample constituents are not known, liquid should never be disposed of down the drain.
 - c. Section 5.1.10/.11 Minimum Chain-of-Custody should include the signing of the Field Chain-of-Custody Record form to acknowledge receipt of the cooler on the COC form. Signing of the COC should never be delayed.

- 10.5. Standard Operating Procedure for Sample Management Procedures (SOP No.: OP SMO-OP, July 15, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

General Comments:

- a. The Weyerhaeuser SOP was issued as a controlled document, with a notation that it was valid for one calendar day from September 16, 2006. The SOP was received by ESAT November XX, 2006, past the valid date of the control number for the SOP. The effective date of the SOP was July 15, 2003. There was no revision number shown on the SOP. There was no indication that the document has been reviewed in the three years since it was issued. The document should be reviewed in accordance with the Weyerhaeuser's quality system, or a frequency recommended by their regulatory agency. A frequency of review/update once every 2 years is recommended in the absence of any other state/ certifying authority requirement. The date of the last revision/review should be noted somewhere in the SOP.

Procedural Review:

- b. Step 3.3.1.f.1 (bullet point 1) states:
"To take a pH of an aqueous sample, mix the sample by inverting the sample container upside down (to get some sample into the lid). Remove the lid and shake liquid from lid onto front rim of the hood, taking care to avoid direct contact with the lid."

Step 3.3.1.f.2 (bullet point 1) states:

"...Remove the lid and shake liquid from the lid onto front rim of the hood, taking care to avoid direct contact with the rim.

Step 3.3.1.f.2 (bullet point 2) states:

"A disposable test tube may also be used, by pouring a very small amount of liquid into the tube. Then dip a pH strip into the tube."

Comment:

The practice of pouring sample directly onto the rim of the hood does not follow the requirements of EPA Method 413.1, is not safe and is likely to result in cross-contamination of samples and possibly spills of hazardous samples. EPA Method 413.1, Step 7.1 states, "...After mixing the sample, check the pH by touching pH-sensitive paper to the cap to ensure that the pH is 2 or lower." Additionally, the Weyerhaeuser SOP does not address clean up of the samples once they have been placed on the rim of the hood.

It should be noted that on April 6, 2004 EPA proposed to withdraw EPA Method 413.1 (69 FR 18165) as an approved method due to its use of an ozone-depleting compound (Freon). When EPA's final rule is approved, Method 413.1 and Standard Method 5520B will be eliminated and only EPA Method 1664A will be approved. If Weyerhaeuser's regulatory permits no longer require the use of EPA Method 413.1, the reference to it and handling samples for analysis by that method should be removed from the SOP.

- c. Step 3.3.1.f.4 states, "Use lead acetate test strips to check for presence of sulfide in liquid samples where cyanide is requested. PbS will precipitate out with color (black). Record pH and sulfide information on Sample Receipt Form)."

Comment:

This statement should be modified to note that if sulfide is present in the sample, it will be indicated by a darkening of the test strip or a dark precipitate on the test strip.

It also should be noted that there is some discrepancy among the reference methods about how lead acetate test strips should be treated prior to use. EPA Methods 335.2 and 335.4 make no mention of any treatment to the test strips prior to use. However, *Standard Methods for the Examination of Water and Wastewater*, Method 4500, Preliminary Treatment of Samples, states that test strips should be moistened with an acetic acid buffer solution prior to adding a drop of sample. The USEPA Region V Central Regional Laboratory SOP for Cyanide requires that the test strips be moistened with 10% H₂SO₄ prior to use. Weyerhaeuser may want to consult the manufacturer of the test strips to determine if the test strips require the use of acid to work properly and then incorporate the requirements into the SOP.

- d. Step 3.3.1.f.5 states, "Check other preservation required as specified, such as RCl₂ as necessary for N. Carolina regulatory samples."

Comment:

The SOP should clearly define the meaning of the abbreviation RCl₂ to ensure that there is no misunderstanding on the part of the reader. It is assumed by the reviewer that RCl₂ is referring to residual chlorine. EPA Methods 335.2, 335.4 and Standard Methods 4500 for the analysis of cyanide require checking samples for the presence of oxidizing interferences, such as residual chlorine, by using potassium iodide (KI) test strips. This requirement should be added to the SOP with the same

level of detail as provided for checking for the presence of sulfide. It would be helpful if there was more detail in this section regarding special regulatory requirements.

- e. Step 3.3.1.g states, " Add any needed preservative in accordance with proper analytical protocol as stated in 40 CFR136.3, Table II.

Comment:

It would be helpful to either add a copy of Table II as an appendix to the SOP or as a controlled document work instruction.

- f. Summary Comments:

In general this SOP seems to be a good, detailed explanation of how samples from several different sources are received and processed into the laboratory. There is sufficient detail to allow personnel to use the SOP as a training document.

The primary issues of concern relate to incorporating sample screening for interferences into the SOP with more detail and resolving the laboratory's practices/requirements for Oil and Grease.

- 10.6. Standard Operating Procedure for Analytical Procedure 3500 (SOP No.: AC G-3500, July 6, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

Series number for some SW-846 Methods have not been updated.

SW-846 5040 should be 5041
SW-846 8010 should be 8011
SW-846 8020 should be 8021
SW-846 8030 should be 8031
SW-846 8040 should be 8041
SW-846 8060 should be 8061
SW-846 8080 should be 8081
SW-846 8090 should be 8091
SW-846 8120 should be 8121

- 10.7. Standard Operating Procedure for Sanitation for Soils and Sediments (SOP No.: AC G-3550, December 7, 1993) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 1.3 refers to SOP 4-P-3540 for GPC Clean-up. No copy of this SOP was provided.
- b. Section 7.2 refers to SOP 1-TS-CLP for calculating percent moisture. No copy of this SOP was provided.
- c. Section 7.4 refers to SOP 4-P-CONC for conducting the extract concentration step. No copy of this SOP was provided.
- d. Section 9.3.1 does not identify the volume of surrogates or matrix spiking solution that should be added to the samples.
- e. Section 9.3.4 does not identify what size or type container the extraction solvent should be decanted into.
- f. Section 9.4.3 does not identify the volume of surrogates or matrix spiking solution that should be added to the samples.

- 10.8. Standard Operating Procedure for Extraction by Continuous Liquid-Liquid Extractor (SOP No.: AC G-3520, April 19, 2004) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 6.4 refers to Millipore water that is not listed in section 5.0 Reagents Required.
- b. Section 6 does not identify how extracts will be stored if they are not immediately analyzed.

- 10.9. Standard Operating Procedure for Polychlorinated Biphenyls Analysis by GC/ECD (SOP No.: AM E-8082, June 20, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. No problems observed.

- 10.10. Standard Operating Procedure for Capillary GC/MS for Semivolatile Organics (SOP No.: AM E-8270, July 18, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 8.2.1 refers to SOP AC G-ORGSTD for preparing organic standards. No copy of this SOP was provided.

- 10.11. MDEQ SOP #213 (Soil Fraction Preparation for Lead Analysis), Revision 2, Effective Date 01/05 was reviewed for technical accuracy and agreement with published references. There is no published EPA Method for the procedure detailed in this SOP. The SOP references listed in Section 21 were reviewed and used as the standard for comparison. Because the purpose of this SOP is to prepare soil samples collected for MDEQ regulatory programs which monitor exposure to lead, the EPA sample preparation method, EPA Method 3050B, was also consulted for sample requirements.

General Comments:

- a. The MDEQ SOP was received as an uncontrolled copy, but was not marked as such. The SOP document cover page should state whether the copy is controlled or not and should contain a control number or be marked as an uncontrolled copy.

Procedural Review:

- b. Step 14.1 states, "Remove all rocks and vegetation from the soil." This statement needs to be more specific since gravelly soil samples are essentially mostly rocks of various sizes. For example-"Remove all rock and vegetation larger than 2 cm in size from the soil.
- c. Step 14.11 states, "...Place the sieved portion into a mortar and grind with a ceramic pestle;" Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must pass through USS Size #10 (2mm diameter particle size). Statement should be revised to say that the sieved portion is ground with a mortar and pestle until all of it passes through a USS Size 10 sieve.
- d. Step 14.11 States, "... (The sieve, mortar, and pestle should be cleaned between samples.)" No specific directions for cleaning these pieces of equipment could be located in the SOP. Must detail method used to clean this equipment (for example-blowing compressed air over it in hood, washing with soap and water, etc.).

- e. Step 14.12 states, " ...Place the portion that did not pass through the sieve into a ceramic pestle mortar and grind with a ceramic pestle;..." Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must be ground sufficiently that they will pass through a USS Size #10 sieve (2 mm).
- f. Step 20.1 states, "Generated waste and samples can be thrown out with the trash if they do not contain identified hazardous material (i.e., samples from an industrial site, etc) that require handling as a hazardous waste." All samples should be handled and disposed of as hazardous waste unless proven otherwise.

SOP Review

Weyerhaeuser submitted six standard operating procedures for review. In addition, Columbia Analytical submitted four SOPs and Michigan DEQ submitted one SOP for review. No guidance was given for how the reviews were to be completed.

Weyerhaeuser

- 11. SOP OP-SMO-OP (Weyerhaeuser Sample Management Procedures SOP) Weyerhaeuser, effective date July 15, 2003, was reviewed for technical accuracy and agreement with published references. There is no published EPA Method for the procedure detailed in this SOP

General Comments:

- a. The Weyerhaeuser SOP was issued as a controlled document, with a notation that it was valid for one calendar day from September 16, 2006. The SOP was received by ESAT Dec. 1, 2006, past the valid date of the control number for the SOP. The effective date of the SOP was July 15, 2003. There was no revision number shown on the SOP. There was no indication that the document has been reviewed in the three years since it was issued. The document should be reviewed in accordance with the Weyerhaeuser's quality system, or a frequency recommended by their regulatory agency. A frequency of review/update once every 2 years is recommended in the absence of any other state/ certifying authority requirement. The date of the last revision/review should be noted somewhere in the SOP.

Procedural Review:

- b. Step 3.3.1.f.1 (bullet point 1) states, "To take a pH of an aqueous sample, mix the sample by inverting the sample container upside down (to get some sample into the lid). Remove the lid and shake liquid from lid onto front rim of the hood, taking care to avoid direct contact with the lid."

Step 3.3.1.f.2 (bullet point 1) states, "...Remove the lid and shake liquid from the lid onto front rim of the hood, taking care to avoid direct contact with the rim. "

Step 3.3.1.f.2 (bullet point 2) states, "A disposable test tube may also be used, by pouring a very small amount of liquid into the tube. Then dip a pH strip into the tube."

Comment:

The practice of pouring sample directly onto the rim of the hood does not follow the requirements of EPA Method 413.1, is not safe and is likely to result in cross-contamination of samples and possibly spills of hazardous samples. EPA Method 413.1, Step 7.1 states, "...After mixing the sample, check the pH by touching pH-sensitive paper to the cap to ensure that the pH is 2 or lower." Additionally, the Weyerhaeuser SOP does not address clean up of the samples once they have been placed on the rim of the hood.

It should be noted that on April 6, 2004 EPA proposed to withdraw EPA Method 413.1 (69 FR 18165) as an approved method due to its use of an ozone-depleting compound (Freon). When EPA's final rule is approved, Method 413.1 and Standard Method 5520B will be eliminated and only EPA Method 1664A will be approved. If Weyerhaeuser's regulatory permits no longer require the use of EPA Method 413.1, the reference to it and handling samples for analysis by that method should be removed from the SOP.

- c. Step 3.3.1.f.4 states, "Use lead acetate test strips to check for presence of sulfide in liquid samples where cyanide is requested. PbS will precipitate out with color (black). Record pH and sulfide information on Sample Receipt Form."

Comment:

This statement should be modified to note that if sulfide is present in the sample, it will be indicated by a darkening of the test strip or a dark precipitate on the test strip.

It also should be noted that there is some discrepancy among the reference methods about how lead acetate test strips should be treated prior to use. EPA Methods 335.2 and 335.4 make no mention of any treatment to the test strips prior to use. However, Standard Methods for the Examination of Water and Wastewater, Method 4500, Preliminary Treatment of Samples, states that test strips should be moistened with an acetic acid buffer solution prior to adding a drop of sample. The USEPA

Region V Central Regional Laboratory SOP for Cyanide requires that the test strips be moistened with 10% H₂SO₄ prior to use. Weyerhaeuser may want to consult the manufacturer of the test strips to determine if the test strips require the use of acid to work properly and then incorporate the requirements into the SOP.

- d. Step 3.3.1.f.5 states, "Check other preservation required as specified, such as RCl₂ as necessary for N. Carolina regulatory samples."

Comment:

The SOP should clearly define the meaning of the abbreviation RCl₂ to ensure that there is no misunderstanding on the part of the reader. It is assumed by the reviewer that RCl₂ is referring to residual chlorine. EPA Methods 335.2, 335.4 and Standard Methods 4500 for the analysis of cyanide require checking samples for the presence of oxidizing interferences, such as residual chlorine, by using potassium iodide (KI) test strips. This requirement should be added to the SOP with the same level of detail as provided for checking for the presence of sulfide.

It would be helpful if there was more detail in this section regarding special regulatory requirements.

- e. Step 3.3.1.g states, "Add any needed preservative in accordance with proper analytical protocol as stated in 40 CFR136.3, Table II. "

Comment:

It would be helpful to either add a copy of Table II as an appendix to the SOP or as a controlled document work instruction.

- a. Summary Comments:

In general this SOP seems to be a good, detailed explanation of how samples from several different sources are received and processed into the laboratory. There is sufficient detail to allow personnel to use the SOP as a training document.

The primary issues of concern relate to incorporating sample screening for interferences into the SOP with more detail and resolving the laboratory's practices/requirements for Oil and Grease.

12. SOP AM E-3010 (Acid Digestion of Aqueous Samples and Extracts):

General comment:

- a. The SOP meets the basic technical requirements for SW846 method 3010.

Suggestion:

The SOP should include detailed information about the spiking solutions used. The SOP should include the concentrations used for each elements and how the solution is prepared. This could be accomplished by a solution formula table. The SOP should also state which analytical method the preparation will be used for. (Example: SW846 6010).

13. SOP AM E-3050 (Acid Digestion of Sediments, Soils, and Sludges):

General comment:

- a. The SOP meets the basic technical requirements for SW846 method 3050.

Suggestion:

The SOP should include detailed information about the spiking solutions used. The SOP should include the concentrations used for each elements and how the solution is prepared. This could be accomplished by a solution formula table. The SOP should also state which analytical method the preparation will be used for. (Example: SW846 6010).

14. SOP AM E-160.2 (Suspended Solids in Water and Wastewater): The method requires the sample to be dried to a constant weight. The SOP indicates in Section 8 that a single dried weight is taken. There is no confirmation that constant weight has been achieved. The SOP lists that a control sample is used. There is no information in the SOP as to what the control is or what limits are being used to indicate the batch is in control. All QC limits should be listed in the SOP

15. SOP AI M-ICPMS (Standard Operating Procedure for the Perkin Elmer ELAN 5000 ICPMS):

General comment:

- a. The SOP is written as a general work instruction for operating the PE ICPMS. The SOP references method 200.8 without giving any detail as to how the instrument is used with the method. Digestion SOPs were submitted for the SW846 series, however, the analytical SOP submitted is

for EPA 200.8. The submitted preparation SOPs may be used for methods 6010 or 6020. If method 200.8 is to be used then the preparation procedure for 200.8 must be followed or method 200.2 should be used. Combining of SW846 methods with EPA methods should not be done.

The SOP should give detail on the tuning of the instrument. The method for checking the tune of the instrument is acceptable. All other technical aspects for the work instruction are acceptable. Individual SOPs for method 200.8 and SW846 6020 should be submitted.

16. SOP AQ M-METALS (Metals Team Supplement QZ/QC Guidelines to the A&T):

General Comment:

- a. The SOP details QC criteria for the variety of metals analytical techniques but never states which criteria goes with which methodology. There are no references between the instrument operating instructions and the QC supplement.

Suggestion:

Separate SOPs should be developed based on each test method number. This will reduce confusion as to which method is being followed due to subtle variations in QC requirements.

Columbia Analytical

17. General comment:

The documents submitted have a box for a Control Number. The box was not completed and no indication was present that the document was an uncontrolled copy.

18. SOP SMO-SCOC (Sample Tracking and Internal Chain-of-Custody): Section 6.5 indicates that, when disposal is performed, each container is scanned. No mention is made regarding how the custody of hazardous samples is handled.

Suggestion:

Include scanning into hazardous waste drum or lab pack.

19. SOP SMO-GEN (Sample Receiving): Section 5.1.8.3 Liquid in coolers from broken samples should be treated as potentially hazardous and disposed of

accordingly. Since sample constituents are not known, liquid should never be disposed of down the drain.

- a. Section 5.1.10/11 Minimum Chain-of-Custody should include the signing of the Field Chain-of-Custody Record form to acknowledge receipt of the cooler on the COC form. Signing of the COC should never be delayed.
20. SOP SMO-SDIS (Sample Disposal): Section 5.2.3.1 indicates that the procedure for disposal of non-hazardous preserved samples is to flush the samples down the drain with large amounts of water. Preserved samples should be neutralized prior to disposal as preserved samples (acidified metals samples and base preserved cyanide samples are hazardous by pH).
- a. Section 5.2.3.2/5.2.3.3 indicates that solid samples considered non-hazardous are disposed of in the garbage. It is not clear how much testing is done on the samples to determine if any hazardous constituents are present. Since there may be hazardous constituents present which were not tested for, solids should be drummed or lab packed and disposed of as potentially hazardous.
 - b. Section 5.2.3.4 does not contain any reference to checks of hazardous constituents other than odor. Foul Condensates should be checked for other hazardous constituents. Also, no check is made to insure that organosulfides are sufficiently neutralized/broken down. The bleach solution pH should then be checked to insure that it is within the correct range for discharge.
 - c. Section 5.2.6.2 indicates that containers for non-hazardous samples are dumped in the normal laboratory garbage. Those which contained preserved samples should be triple rinsed and the rinsate neutralized to insure no preservative residue is present in the bottles.
 - d. Section 5.2.6.3 indicates that glass containers which contained hazardous samples are included in the sample waste handling but that plastic containers are dumped in the normal laboratory garbage. Plastic containers should at least be triple rinsed and the rinsate treated as hazardous liquid waste or the plastic bottles treated as hazardous solid waste.
21. SOP MET-1631 (Mercury in Water by Atomic Fluorescence Spectrometry): The SOP meets the technical requirements for EPA method 1631.

MDEQ

MDEQ SOP #213, Revision 2, Effective Date 01/05 was reviewed for technical accuracy and agreement with published references. There is no published EPA Method for the procedure detailed in this SOP. The SOP references listed in

Section 21 were reviewed and used as the standard for comparison. Because the purpose of this SOP is to prepare soil samples collected for MDEQ regulatory programs which monitor exposure to lead, the EPA sample preparation method, EPA Method 3050B, was also consulted for sample requirements.

22. General Comments:

The MDEQ SOP was received as an uncontrolled copy, but was not marked as such. The SOP document cover page should state whether the copy is controlled or not and should contain a control number or be marked as an uncontrolled copy.


23. Procedural Review:

- a. Step 14.1 states, "Remove all rocks and vegetation from the soil." This statement needs to be more specific since some soil samples may be mostly rocks of various sizes. For example-"Remove all rock and vegetation larger than 2 cm in size from the soil.
- b. Step 14.11 states, "...Place the sieved portion into a mortar and grind with a ceramic pestle;" Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must pass through a USS Size #10 (2mm diameter particle size) sieve. Statement should be revised to say that the sieved portion is ground with a mortar and pestle until all of it passes through a USS Size 10 sieve or equivalent.
- c. Step 14.11 States, "... (The sieve, mortar, and pestle should be cleaned between samples.)" No specific directions for cleaning these pieces of equipment could be located in the SOP. The SOP must detail the method used to clean this equipment (for example-blowing compressed air over it in hood, washing with soap and water, etc.).
- d. Step 14.12 states, "Place the portion that did not pass through the sieve into a ceramic pestle mortar and grind with a ceramic pestle;" Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must be ground sufficiently that they will pass through a USS Size #10 sieves (2 mm).
- e. Step 20.1 states, "Generated waste and samples can be thrown out with the trash if they do not contain identified hazardous material (i.e., samples from an industrial site, etc) that require handling as a hazardous waste." It is not clear how much testing is done on the samples to determine if any hazardous constituents are present. Since there may be hazardous constituents present which were not tested for, solids should be drummed or lab packed and disposed of as potentially hazardous.

Please note that these recommendations do not constitute Region 5's comments to the *Work Plan* or *Sampling and Analysis Plan*.

Thank you for your attention to this matter. Please do not hesitate to call me at 312.886.1434, should you have any questions related to the task.

Sincerely,



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